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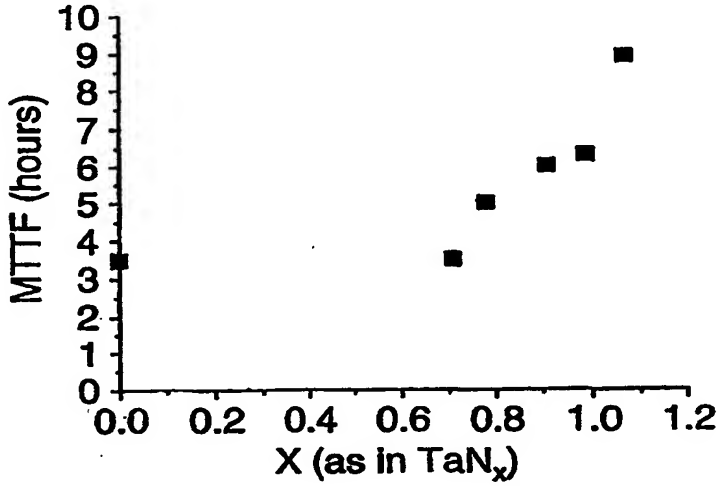
(54) Title: CONTINUOUS PROCESS FOR SPUTTERING TANTALUM NITRIDE FILMS



(57) Abstract

The barrier properties of PVD deposited tantalum nitride barrier films on a substrate are improved by biasing the substrate during deposition and wherein the bulk of the barrier film is TaN_x wherein x is less than or equal to 1 and the barrier film has a resistivity of up to about 500 microohms–cm. A continuous method for depositing tantalum nitride layers on successive substrates stabilizes a flow of argon gas alone in the chamber, sputter deposits a first layer of tantalum or a tantalum–rich layer, then adds nitrogen to deposit tantalum nitride while biasing the substrate and maintaining a high nitrogen content in the film, and, by shutting off the flow of nitrogen, deposits tantalum or a tantalum–rich film to remove any tantalum nitride deposits on the surface of the tantalum target. This method avoids hysteresis drift of the composition and properties of the barrier films over successive depositions.

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CONTINUOUS PROCESS FOR SPUTTERING TANTALUM NITRIDE FILMS

This application is a continuation-in-part of copending application Serial No. 09/058,432 filed April 10, 1998.

This invention relates to the deposition of improved barrier layers for copper metal lines and vias for the manufacture of semiconductor devices. More particularly, this invention relates to depositing tantalum-containing barrier layers having enhanced barrier performance.

BACKGROUND OF THE INVENTION

In the manufacture of semiconductor devices, conductive metal contacts and lines are deposited over dielectric layers, such as silicon oxide. Heretofore aluminum has been the metal conductor of choice. Since aluminum diffuses into silicon during elevated temperature processing, a barrier layer, particularly one including titanium nitride, is conventionally deposited between the substrate and the aluminum to prevent diffusion or "spiking" by the aluminum into the substrate.

Copper is a better conductor than aluminum, and it has a higher resistance to electromigration than aluminum. However, copper also diffuses into silicon as well as many other materials under conditions of elevated temperature and applied electric field. Thus a good barrier layer is also essential for copper lines and vias.

Tantalum has been tried as a barrier layer for copper. Tantalum is a good conductor and a good wetting agent for overlying copper layers. Tantalum nitride, formed by sputter depositing tantalum in the presence of nitrogen gas, is a better barrier than tantalum alone, but it has a higher resistivity. In order to take advantage of the excellent conductivity of copper, a barrier layer of tantalum and/or a tantalum nitride barrier layer, which is much less conductive, must be as thin and as conformal as possible.

Conventional sputtering, particularly into small diameter, high aspect ratio openings, has been found to be inadequate to deposit thin conformal coatings into such openings. Conventional sputtering is carried out in a high vacuum chamber using a target of the material to be sputtered, which is connected to a source of DC power. A substrate is mounted on a support that is spaced from and parallel to the target. Argon is passed into the chamber. The negative voltage on the target attracts argon ions to the target surface after it is powered, where these argon ions impact and sputter off particles of the target material. These sputtered particles then deposit on the substrate.

Unfortunately however, sputtering does not occur only in the vertical direction, but in all directions except the horizontal. Thus when high aspect ratio, small diameter

openings are to be filled, fewer sputtered particles deposit on the bottom and sidewalls of the openings than on the top of the openings. This is illustrated in Fig. 1 which illustrates the buildup of target material 10 on the top 12 and upper sidewalls 14 of a high aspect ratio opening 16. This buildup prevents many sputtered particles from reaching the bottom 18 and the bottom sidewalls 20 of the opening 16. Thus the resultant coating, as of a barrier material, is not as conformal as is required.

In order to improve the directionality of sputtered particles, an improved sputtering chamber has been developed. A high density plasma is formed in a sputtering chamber between the target and the substrate by means of an inductive coil coupled to a source of RF power. As particles are sputtered from the target, they pass through a plasma region in the vicinity of the coil and become ionized in this region. When the substrate is biased, as by powering the substrate support, the substrate has a negative potential and the positively charged sputtered ions are attracted to the substrate. They then impact the substrate in a highly perpendicular direction. Thus when using this improved sputtering chamber, more of the sputtered particles deposit on the bottom and bottom sidewalls of high aspect ratio openings, significantly enhancing the bottom coverage and leading to

more conformal sputtered layers.

This improved sputtering chamber is known as an "ionized metal plasma" or "IMP" chamber, as shown in Fig. 2. The IMP chamber 170 includes a conventional target 172, as of tantalum, mounted on a top wall 173 of the chamber 170. A pair of opposing magnets 176, 178 are mounted over the top of the target 172. A substrate support 174, bearing a substrate 175 thereon, is mounted opposite to the target 172. A source of power 180 is connected to the target 172 and a source of RF power 182 is connected to the substrate support 174. A controller 200 regulates gas flows. A helical coil 186, which can have one or more turns, is preferably made from the same material as the target 172. The coil 186 is mounted between the target 172 and the substrate support 174, and is also connected to a source of RF power 188. Gases such as argon and nitrogen in vessels 192, 194, are metered to the chamber 170 by means of flow valves 196, 198 respectively.

Providing that the pressure in the chamber is fairly high, i.e., from about 10 millitorr up to 1 torr, the internal inductively coupled coil 186 provides a high density plasma in the region between the target 172 and the support electrode 174. If the pressure is too low, too few particles are present and sufficient metal ionization will not occur in the region of the powered coil. A gate valve 199 is used to regulate the

pumping speed and regulate the pressure in the chamber 170 to the desired range of about 10 millitorr up to 1 torr.

Although the use of the IMP chamber has resulted in the deposition of more robust barrier layers, and better conformality of the barrier layers, various problems remain. Improvements continue to be sought in the method of deposition to improve tantalum-containing barrier layers for copper lines and vias, and to ensure that a continuous process can produce improved barrier layers on successive substrates having uniform properties and long device life.

SUMMARY OF THE INVENTION

We have found that tantalum nitride barrier layers having improved density and film smoothness are obtained when bias is applied to the substrate support during deposition. We have also found that the concentration of nitrogen in tantalum nitride films has a large effect on the robustness of tantalum-containing barrier layers. The tantalum nitride barrier performance improves with increasing nitrogen content in the tantalum nitride film. However, we have also found that there is a limit to the amount of nitrogen that can be added without dramatically increasing the resistivity of the films.

Still further, we have found that using a continuous process for the successive manufacture of substrates, some hysteresis drift occurs that in turn leads to a drift in film

resistivity and thickness. We have found this hysteresis drift can be prevented by carrying out the following sequential steps during sputter deposition of tantalum-containing barrier layers:

a) stabilizing a gas flow of argon in the absence of nitrogen in the sputtering chamber;

b) igniting a plasma in the chamber, either using the stabilized argon alone or introducing a low level of nitrogen to the stabilized argon;

c) depositing tantalum nitride from a stabilized mixture of argon and an amount of nitrogen at least equal to the amount introduced in step b); and

d) turning off the flow of nitrogen while continuing sputtering to clean the tantalum target.

Following these sequential steps, sequential layers of a first layer of a tantalum or tantalum-rich tantalum nitride film, a second tantalum nitride film and a final tantalum or tantalum-rich tantalum nitride film are deposited on the substrate. After stabilization of argon alone, the initial sputter deposition may be carried out in argon alone, to form a tantalum film, or a low level of nitrogen (generally less nitrogen than is required to form a TaN film) can also be introduced into the argon to form a tantalum-rich film. The nitrogen gas flow is then introduced into the chamber to

deposit a tantalum nitride film. By shutting off the flow of nitrogen as the last step of the deposition cycle, the target is cleaned to remove nitrogen-containing material prior to the beginning of the next deposition cycle. By carefully regulating the gas flows of argon and nitrogen, the deposited tantalum-containing barrier films remain uniform in composition and barrier characteristics from one substrate to another.

BRIEF DESCRIPTION OF THE DRAWING

Fig. 1 is a cross sectional view of an opening partially filled with material in accordance with prior art processes.

Fig. 2 is a schematic cross sectional view of a modified physical vapor deposition chamber useful in the present invention.

Fig. 3 is a schematic cross sectional view of a test MOS capacitor.

Fig. 4 is a graph of a TaN hysteresis curve of voltage versus nitrogen flow showing a rapid increase in the target voltage when the poison mode is reached.

Fig. 5 is a graph of resistivity and deposition rate versus nitrogen flow rate for tantalum nitride films.

Fig. 6 is a graph of MTTF in hours versus the value of x in TaN_x.

DETAILED DESCRIPTION OF THE INVENTION

The robustness of a tantalum-containing barrier layer for copper lines and vias can best be characterized by electrical testing of MOS test capacitors having a structure Si/SiOx/TaN barrier/Cu using a bias temperatures stress test (BTS) at 275°C and 2MV/cm.

A typical metal-oxide semiconductor (MOS) test capacitor is shown in Fig. 3. A silicon substrate 30 is covered with a dielectric layer 32, such as silicon oxide, generally about 1000 angstroms thick. A thin barrier layer 34 is deposited and a conductive metal layer 36 is deposited over the barrier layer. The current through the oxide layer is measured. When copper diffuses through the barrier layer into the oxide layer, a catastrophic failure of the oxide is noted. The median time to failure (MTTF) is used to evaluate the barrier performance. The failure distribution gives the standard deviation of the data points.

We have found that tantalum and tantalum-containing barrier films deposited by IMP technology have a very smooth surface morphology. This is true for deposition with or without wafer bias.

Surface roughness of various films was measured by scanning probe microscopy. As a comparison, typical silicon films have RMS values, i.e., the standard deviation of Z values within a given area, of 0.8-0.9 angstroms. Silicon

oxide #10 has an RMS value of 1.6. Measured surface roughness for a 2 micron x 2 micron area near the center of each substrate for TaN films about 250 angstroms thick is given in the Table below.

The biased substrates were biased using 350 watts bias in an IMP chamber using a 1 kw DC power source and 1.5 kw of RF power. The chamber was held at 22.5 millitorr pressure for tantalum films, and at 28 millitorr pressure for tantalum nitride films. RMS values are given in the Table below:

TABLE

Material	RMS
Ta, no bias	2.2
Ta, with bias	2.5
TaN, no bias	2.0
TaN, with bias	1.5

Thus using an IMP chamber to deposit tantalum or tantalum nitride, the surface of the film was almost as smooth as the silicon oxide which underlies the tantalum-containing or tantalum nitride barrier films. Thus no surface roughness is added to the device structure by the present tantalum-containing layers using an IMP deposition technique.

The density of tantalum nitride films is increased by biasing during deposition. The resultant barrier layers are amorphous and dense, further contributing to the utility of the present films for making barrier layers that do not permit

diffusion of an overlying metal layer, such as copper, through the barrier layer. This was shown in a comparison between tantalum nitride layers deposited without bias and with bias. The density and nitrogen content were measured by Rutherford Backscattering. When no bias was used during deposition, a $TaN_{0.85}$ film had a density of 8.3 atoms/cm^3. When bias was used during deposition, a $TaN_{0.85}$ film had a higher density of 9.2 atoms/cm^3. In general, an increase in the density of tantalum nitride films of about 9% is found when biasing is used during tantalum nitride sputter deposition.

Increasing the nitrogen content of tantalum nitride films also increases the density of the films, which in turn improves barrier performance.

We have also found that barrier performance is linked to the nitrogen content of the tantalum nitride (TaN_x) films. The amount of nitrogen in the tantalum nitride increases as the flow of nitrogen in the IMP chamber increases. The resistivity of the films remains fairly constant until a threshold level is reached, generally when x in TaN_x is about 1. Above the threshold level, the resistivity increases dramatically, as shown in Figs. 4 and 5. At that point the deposition rate also decreases rapidly, as shown in Fig. 5.

Barrier performance was tested using MTTF as a criterion for nitrogen gas flows in an IMP chamber of 16, 18 and 20

sccm, when the MTTF was 2.4, 3.4 and 6.0 hours respectively. A dielectric TaN_x film wherein x>1 was also a good diffusion barrier, but of course the presence of the dielectric tantalum nitride increases the resistivitvy of the contact after deposition of copper. Fig. 6 is a graph of MTTF versus the nitrogen concentration in TaN films illustrating an increase in MTTF with an increase in nitrogen concentration.

Thermal anneal tests were also used to evaluate tantalum nitride performance. Film samples of tantalum nitride 100 angstroms thick containing various amounts of nitrogen on silicon substrates, with a 1500 angstrom thick layer of copper thereover, were thermally stressed at 600°C in a PVD chamber for 15 minutes. Sheet resistance and film appearance were compared before and after the anneal. Tantalum nitride having greater than about 30 atomic percent of nitrogen displayed no increase in resistivity after annealing. Secco etching was then used to remove the copper and the tantalum nitride layers, exposing the silicon surface. No etch pit formation was apparent, indicating good barrier performance, which improved with increasing nitrogen concentration up to about stoichiometric tantalum nitride layers.

Thus the ideal nitrogen content of tantalum nitride barrier layers is the maximum nitrogen level that is below the threshold level when the resistivity increases rapidly, as

shown in Fig. 4.

However, it is difficult to achieve this balance, particularly in a high production environment wherein successive depositions are performed in the same chamber on a continuous basis. We have noted that if nitrogen is added to argon during the first step of the present process, over time hysteresis drift occurs which in turn can lead to poison-mode operation which increases the resistivity of the tantalum nitride film and lowers the deposition rate. This in turn causes a drift in film composition, film resistivity and thickness uniformity.

This hysteresis drift can be eliminated by shutting off the nitrogen flow entirely at the beginning of the tantalum nitride deposition, e.g., during the stabilization and ignition of a plasma in an IMP chamber, and at the end of the deposition. By sputtering a tantalum target in the absence of nitrogen for a few seconds at the end of each deposition cycle, any tantalum nitride that has formed on the target will be sputtered off, presenting a clean, non-poisoned target for each new substrate admitted to the chamber. This is true also of the tantalum coil used in the IMP chamber.

To illustrate this, a tantalum nitride film over 10 microns in thickness (equivalent to over 250 depositions) was sputtered while turning off the flow of nitrogen in the

chamber during the initial step of the present process. No increase in the resistivity of the deposited tantalum nitride film was noted. However, if the nitrogen flow is maintained at a uniform level throughout sequential depositions, over time the resistivity of the tantalum nitride layer on each substrate becomes higher, and eventually a dielectric tantalum nitride layer is deposited.

To further demonstrate the above continuous process, TaN_x layers were successively deposited to a thickness of about 250 angstroms on 9000 successive silicon dioxide coated wafers in an IMP chamber. The resistivity values of the layers remained the same within a 2% deviation, indicating excellent repeatability. The particle level in the IMP sputtering chamber remained very low even after processing 9000 consecutive wafers. The tantalum nitride coverage at the bottom of openings 0.25 micron in diameter and 1.4 micron deep (aspect ratio of over 5.5:1) was uniform throughout.

The high nitrogen content TaN_x films deposited in accordance with the invention are dense and amorphous; thus few pathways for diffusion of copper through these barrier layers exist.

Other advantages to this continuous process are that the deposited tantalum-containing film is tantalum-rich at both the interface with the substrate and at the interface with the

overlying copper layer. The thickness of these layers can be tailored at the beginning and at the end of the deposition process. The adhesion of these layers is improved, and the texture of the films is also improved. This in turn improves electromigration resistance and hence the overall reliability of the interconnect. A tantalum-rich film at the overlying copper interface may also improve copper nucleation when CVD copper layers are deposited, since a typical CVD copper deposition is based upon an electron transfer, disproportionation reaction.

The present process thus provides conformal step coverage, even in very high aspect ratio openings, and process flexibility in which the nitrogen content in TaN_x layers, deposition pressure, temperature and bias power can be varied.

Although applicants have described the present invention terms of specific embodiments, one skilled in the art will recognize that various changes can be made to the deposition method of tantalum nitride films which are meant to be included within the scope of the appended claims. For example the improvement in properties of tantalum-containing films having a high nitrogen content, deposited while biasing the substrate, will be had whether the tantalum nitride is deposited in an IMP chamber or in a conventional PVD chamber. Thus the invention is meant to include the use of additional

reaction chambers and method steps and is only meant to be limited by the scope of the appended claims.

We Claim:

1. A method of forming a barrier layer for copper lines and vias on a semiconductor substrate comprising

sputtering a tantalum target in a physical vapor deposition chamber in a flow of nitrogen gas sufficient to deposit a TaN_x film wherein x is less than or equal to one, and the film resistivity is at or below about 500 microohms-cm.

2. A method according to claim 1 wherein the film resistivity is at or below about 350 microohms-cm.

3. A method according to claim 1 wherein said physical vapor deposition chamber includes an internal electrode connected to a source of RF power to form a plasma in the chamber between the target and a support for said substrate spaced from and parallel to said target.

4. A method according to claim 1 wherein said substrate is biased during deposition.

5. A continuous method of depositing tantalum-containing barrier films on successive semiconductor substrates by physical vapor deposition which comprises

a) stabilizing a gas flow of argon alone in the deposition chamber,

b) sputtering a first layer of tantalum, using argon alone, or adding a small amount of nitrogen to the argon to form a tantalum-rich tantalum nitride film from a tantalum target in the chamber;

c) adding a flow of nitrogen sufficient to deposit a tantalum nitride (TaN_x) film wherein x is less than or equal to 1 and the film resistivity is at or below about 500 microohm-cm to a desired thickness,

d) shutting off the flow of nitrogen and continuing to sputter the tantalum target to remove any TaN_x deposits on the target, and

e) repeating steps a) to d).

6. A method according to claim 5 wherein said physical vapor deposition chamber includes an internal electrode connected to a source of RF power to form a plasma in the chamber between the target and a support for said substrate spaced from and parallel to said target.

7. A method according to claim 5 wherein said substrate is biased during deposition.

8. A method according to claim 5 wherein the resistivity of the film formed in step b) is at or below 350 microohms-cm.

9. A method of depositing TaN barrier films on a semiconductor substrate by physical vapor deposition which comprises

a) stabilizing a flow of argon alone in a physical vapor deposition chamber;

b) igniting a plasma to deposit a tantalum film or depositing a tantalum-rich tantalum nitride film from a mixture of argon and a small amount of nitrogen on the substrate,

c) adding nitrogen to the chamber in an amount so as to form a tantalum nitride film having a maximum amount of nitrogen but below that amount of nitrogen that causes the resistivity of the TaN film to rise above about 500 microohms-cm, and

d) shutting off the nitrogen flow and continuing to deposit tantalum until nitrogen-containing material has been removed from the tantalum target.

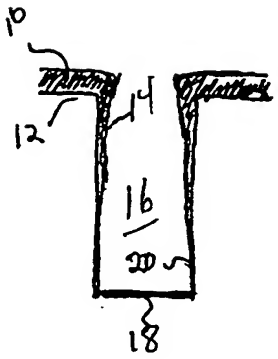

FIG. 2

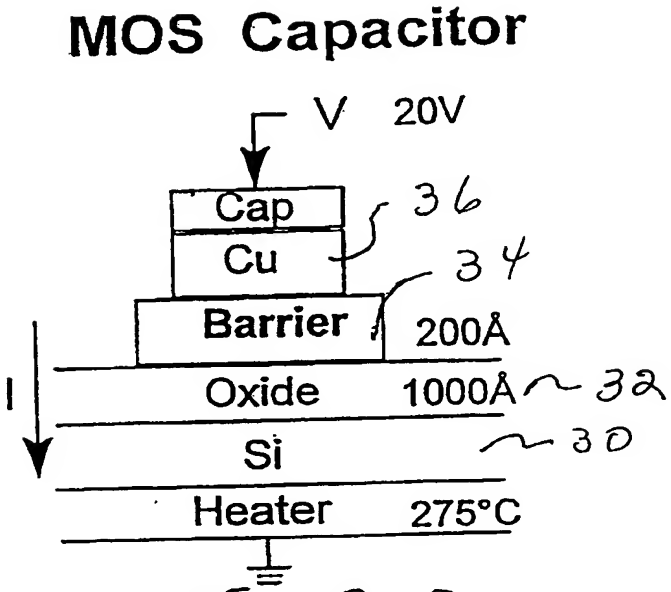

FIG. 3

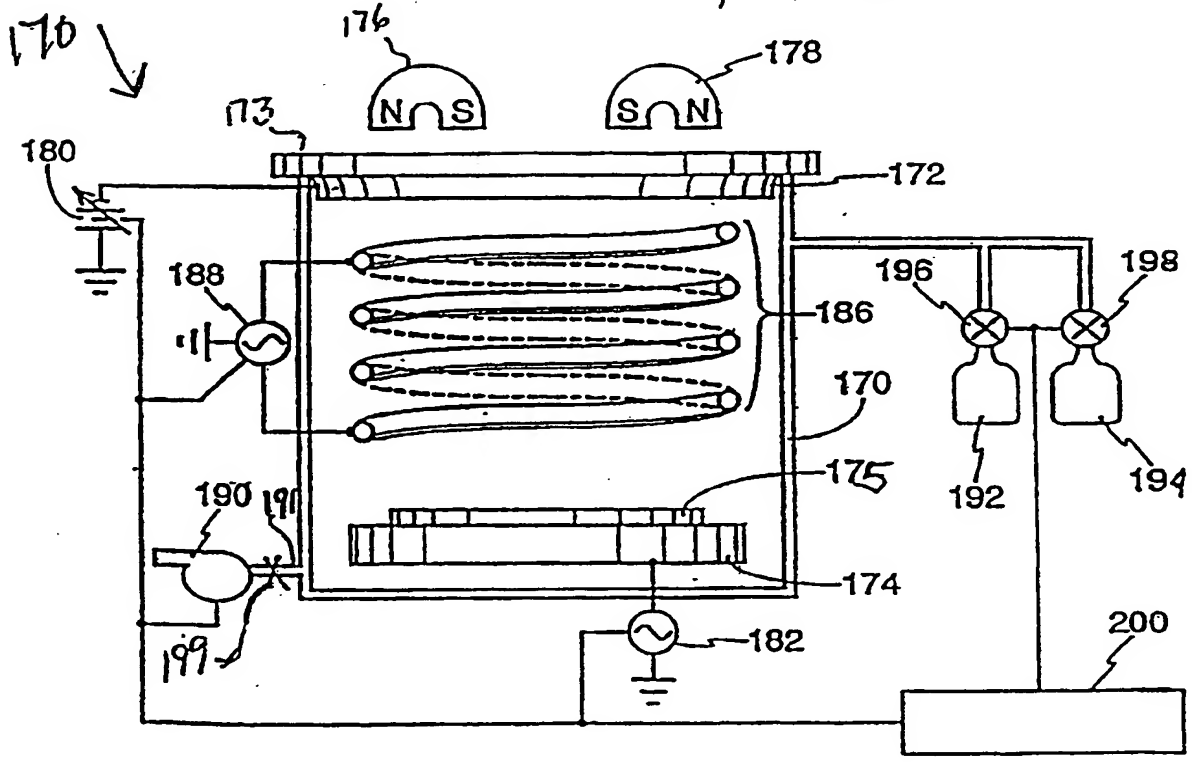

FIG. 2

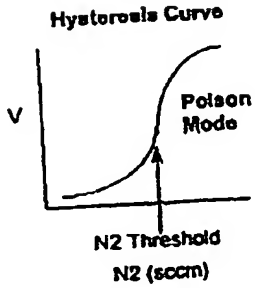

FIG. 4

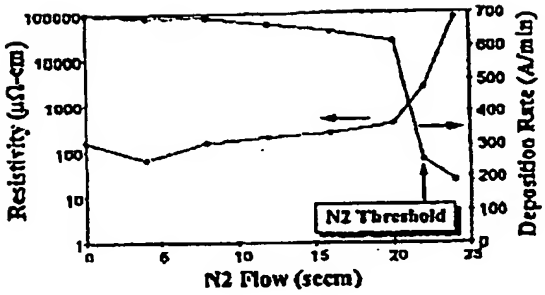

FIG. 5

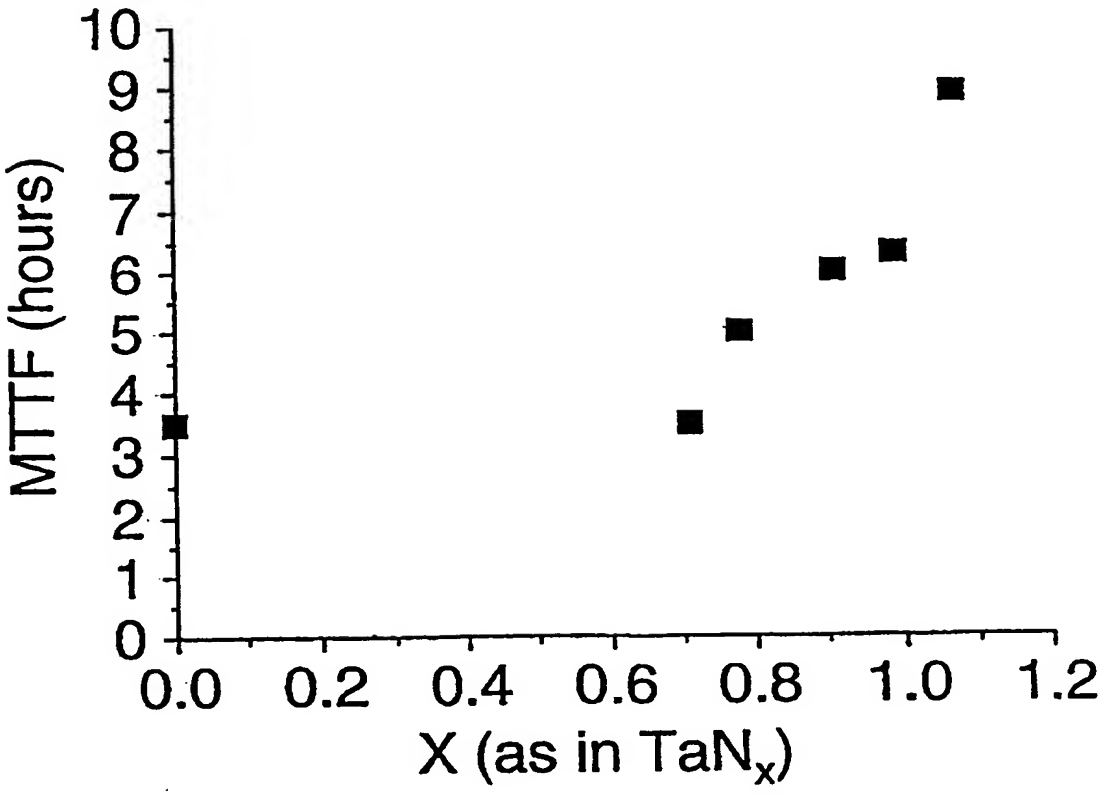

FIG. 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/18178

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : C23C_14/34
US CL : 204/192.12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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APS (USPTO and JPO)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,281,485 A (COLGAN et al.) 25 January 1994, col. 5 lines 47-53, col. 6 lines 29-33 and 60-65,	1, 2, 5, 8, 9
Y	US 5,707,498 A (NGAN) 13 January 1998, col. 1 lines 56-60, col. 3 lines 42-45, col. 4 lines 7-12 and 32-34	3, 4, 6, 7
A,P	US 5,783,282 A (LEIPHART) 21 July 1998	1, 4, 5, 7, 9
Y	U.S. 4,976,839 A (INOUE) 11 December 1990, col. 6 lines 40-43, col. 7 lines 23-24	1, 2, 5, 8, 9
Y	U.S. 5,725,739 A (HU) 10 March 1998, col. 2 line 66 - col. 3 line 12, col. 8 lines 5-10, col. 8 lines 23-26	1, 4, 5, 7

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search 30 DECEMBER 1998	Date of mailing of the international search report 26 JAN 1999
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer JULIAN A. MERCADO Telephone No. (703) 308-0661

Form PCT/ISA/210 (second sheet)(July 1992)*

INTERNATIONAL SEARCH REPORT

International application No.
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B. FIELDS SEARCHED
Minimum documentation searched
Classification System: U.S.

204/192.12, 192.11, 192.15, 192.22, 192.25, 192.17, 298.06, 298.13, 298.12